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NASA CONTRACTOR TECHNICAL REPORT

CONTRACT NAS8 - 31605

EXTENDED ANALYSIS OF SKYLAB EXPERIMENT M558 DATA

By

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June 1976

FINAL REPORT

Prepared for

**NATIONAL AERONAUTICS AND SPACE ADMINISTRATION
GEORGE C. MARSHALL SPACE FLIGHT CENTER
MARSHALL SPACE FLIGHT CENTER,
ALABAMA 35812.**

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SKYLAB EXPERIMENT M558 DATA Final Report
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SUMMARY

This is the Final Report on NASA Contract NAS8-31605, Extended Analysis Of Skylab Experiment M558 Data. A careful review of the data from Skylab M558 has been made in an effort to explain the apparent anomaly of the existence of radial concentration gradients whereas none should have been observed. The very close modelling of the experimental axial concentration profiles by the unsteady-state one-dimensional solution of Fick's Law of self-diffusion in liquid zinc, and the condition of initial uniform concentration in the radioactive pellet portion of the experimental specimens would have precluded the appearance of such radial concentration gradients.

A second and critical look has been taken under the contract NAS8-31605 at the Oak Ridge National Laboratory (ORNL) sample-preparation and data-taking procedure, container effects, the effects of the procedure for bonding Zinc-65 to Zinc rods, and at surface phenomena such as surface tension in an effort to better explain the observed radial concentration gradients. Statistical analyses were used to test the significance of the observed deviation from radial-concentration homogeneity. A student t - distribution test of significance showed that, at 90% or even at 80% level of significance, there were no significant deviations from uniformity in radial concentrations. It was also concluded that the most two likely causes of any deviation that existed were the zinc to zinc-65 bonding procedure and surface phenomena such as surface-tension and capillary action.

Therefore, the following have been recommended:

1. Until additional experimentation has been carried out, the observed radial-concentration gradients should be regarded as of no great significance. It could have been caused by statistical data scatter.
2. Further ground-based experiments should be performed to isolate the effects of bonding procedure.
3. If a flight opportunity occurs, experiments should be flown with an aim to identify the effects of surface tension and sample configuration similar to that of Skylab M558 samples.

INTRODUCTION

Deviation from homogeneity was observed in the radial distribution of radio-active zinc in the Skylab M558 experiment (1). The present study under contract NAS8-31605 was carried out as a review of the data from the M558 experiments in an attempt to pin down the true explanation of the phenomenon observed. The radial distribution of radioactive zinc-65 tracer which had been observed in the M558 experiments had not been satisfactorily explained by initial radioactive zinc distribution in the samples used in the experiments, not by convection in liquid zinc nor by zero-gravity environment alone. Yet the observed distribution had been too pronounced to be ignored.

Skylab M558 experiment measured self-diffusion coefficient of radioactive zinc in liquid non-radioactive zinc in the micro-gravity environment of Skylab in Outer Space. The data obtained therefrom had been modeled closely in the axial direction by Fick's law of diffusion. According to Fick's law, the geometry of the samples and the initial distribution of radioactive zinc tracer in the samples, there should have been uniform radial radioactive zinc distribution. There should have been no radial concentration gradients. The gradients should have existed in the axial direction only. However marked concentration gradients were observed on all three zinc ampoules returned from Skylab. All attempts in this 2½ month study to find, for the observed data, explanations more plausible than those already advanced by the principal investigator of experiment M558 (Ref.1) and by the scientists at Oak Ridge National Laboratory who fabricated the samples have not completely resolved the issue.

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PROCEDURE

The review of the M558 data was performed in the following sequence:

- (1) Review of Oak Ridge National Laboratory (ORNL) Sample preparation and data-taking procedures for sources of error.(2)
- (2) Container effects on the Zn-65 distribution.
- (3) Effects of the Zn-65 to Zn bonding procedure.
- (4) Error analysis on the Skylab M558 data in order to determine whether measured radial inhomogeneities can be accounted for by scatter in the data.
- (5) Evaluation of the results of the foregoing studies to determine whether they adequately explained the Zn-65 radial distributions.
- (6) Consideration of any other factors which might have influenced the radial distribution observed.
- (7) Recommendation for or against further studies possibly involving computer modeling of the mass transport evidenced in the Skylab samples, including possible convective effects.

Equipment

No new equipment was allowed for under the contract NAS8-31605 and none was purchased. None was found necessary within the scope of work of this contract. However a digital computer was used to run short programs to solve Fick's law of diffusion equation and to make statistical error analyses.

1. Review of ORNL Sample Preparation and Data-taking Procedure for Sources of Error

The scientists at ORNL who prepared the samples and sectioned them for data taking, (D. N. Braski, E. H. Kobisk, et. al.) were contacted about their procedure. They had nothing new to add to what they had reported in Ref. 2 and 3 about their procedure. Their data-taking procedure may be summarized as follows:

Before a sample was sectioned for activity counting, its overall activity was measured in a gross ionization chamber; its length and average diameter was measured using a direct reading caliper, and its weight was determined using a Mettler M-5 microbalance.

For precise sectioning and counting of activity, the sample was machined into approximately fifty 0.10-cm (0.040-in.)-thick transverse sections which were accurately indexed. An Edelstaal Unimat jeweller's lathe with ultrasharp tool bit was used to cut the transverse sections into shavings which were quantitatively collected and heat sealed in preweighed plastic envelopes. Each envelope containing zinc sample shavings was subsequently reweighed to determine the zinc weight by difference. Every sixth or seventh section was machined off into three approximately equal parts by weight in radial portions. The three radial sections were labelled "outer" (O), "middle" (M), and "inner" (I) radial sections (Appendices A,B,C of Ref.2). The gamma intensity of each envelope of zinc shavings was measured using a low-level gamma-ray spectrometer coupled with a Nuclear Data 4420 mini-computer system for rapid data collection, analysis and storage. The intensity in micro-curie was divided by weight in grams to get the concentration of the section in $\mu\text{Ci/gm}$. The concentration was then plotted against the location of the section on the zinc cylinder.

Table 1 summarizes the over-all losses in weight and radioactivity due to lost zinc shavings.

TABLE 1

Zinc Cylinder Data From Skylab M558

Capsule No.	A6	A7	B5
Zn Wt before sectioning (gm)	13.5807	13.6349	13.3650
Zn Wt After Sectioning (gm)	13.1595	13.2675	13.0177
% Wt Loss to Sectioning	3.1	2.7	2.7
Activity Before Sectioning (μCi)	8.34	7.75	7.32
Activity After Sectioning (μCi)	7.66	6.699	6.223
% Activity loss to Sectioning	8.1	13.5	15.0

Discussions with the investigators at ORNL revealed the following:

1. The samples had been destroyed as radioactive waste soon after the final report was written on M558. After all the samples had been destructively tested and the activity had decayed to very low levels in the intervening time after the final report had been in. Therefore no useful purpose would be served in preserving the cut-up samples. The loss of the samples meant that no additional check could be made on the accuracy of the measurements reported.

2. Great care had been taken to measure the data accurately. The weighing was accurate to 10^{-4} gm and the activity could be measured to $5(10^{-4})$ μ Ci. It was also pointed out that since only the weights and radioactivities of those samples not lost to sectioning were actually used in determining the concentration (μ Ci/gm), it was not important that some small amount had been lost. The activities and the weights of the lost dust were not included in the data reported; thus the data had been normalized anyway.

3. A careful look at the radial data given in Fig 20 to 22 revealed that nearly all of the observed radial distribution anomalies occurred near or at the Zn-65 and ordinary Zn bonding line in all three Skylab samples tested. This phenomenon would tend to rule out causes due to error in data analysis by sectioning. It would rather tend to point to effects of the method of bonding of the Zn-65 to the Zn pellet, and also to surface and heat-transfer phenomena.

2. Container Effects on the Zn-65 Distribution

During the fabrication of the Skylab M558 samples, the Zinc rod with its bonded Zn-65 pellet was surrounded with a graphite sleeve so as to prevent liquid zinc from wetting and sticking on to the enclosing tantalum tube on solidification. However, since the graphite sleeve was porous, there was the opportunity for the liquid near the surface to travel by capillary attraction through the pores and by surface tension up the wall of the ampoule. Such a surface phenomenon could account for the sudden high concentration of tracer found near the surface next to the graphite walls. In addition, the small space tolerance between the zinc rod and the graphite walls which had been put in to allow for volume expansion on melting, could also cause an initial forcing of liquid zinc up between the graphite wall and the still-solid zinc. The sudden increase in volume on melting meant that the solid zinc was heavier than the liquid zinc with the result that solid zinc would slowly sink into the liquid zinc. Minor perturbations and displacement of radioactive zinc would occur. However, this last argument does not adequately explain the observation that the radial anomalies occurred in all three samples representing the three cases, Zn-65 melting first, in-between, and last.

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If this argument were adequate, then the results should have been more pronounced in the case where Zn-65 pellet melted first than in the case where it melted last (Fig. 22 and 20, respectively, Ref. 1). Therefore, we believe that laboratory experimentation and simulation should be performed to test these theories of surface and container effects. Unfortunately, the present contract did not allow for such a detailed accurate analysis.

3. Effects of Method of Bonding Radioactive Zinc to Ordinary Zinc

A careful review of the procedure for bonding the radioactive zinc-65 pellet to the non-radioactive zinc rod in the fabrication of the samples has led us to strongly believe that it could have been a major contributor to the observed radial anomalies. Briefly, the samples were bonded according to the following procedure.

A 5-ton capacity hydraulic press and a hardened steel die with a 0.72-cm-diam ram (zinc specification) were used to cold-join the specimens. To join A-type specimens (Fig. 1a), the mating surfaces of the Zinc-65 pellet and a 4.04-cm-long rod of zinc were first lightly abraded on 600-grit emery paper and then etched in acid solution. The zinc bar was then inserted in the die on top of the bottom ram with the abraded surface upwards. The Zinc-65 pellet was then inserted in the die, followed by the upper ram, and the entire assembly was placed in the hydraulic press. A compression pressure at the zinc interface of $1.61337(10^5)$ KN/m² to $1.83400(10^5)$ KN/m² (that is 23,400 psi - 26,600 psi) generated by the press was sufficient to produce a cold-welding bond between the zinc bar and zinc-65 pellet. The B-type specimens (Fig. 1b) were joined in a similar manner, except that two bonding operations were needed. The second bonding required a higher compressive pressure to effect bonding due to work hardening of the pellet from the first bonding operation. When the bonding process failed during the first operation, a higher pressure was again needed to effect bonding during subsequent operations, as a result of work hardening.

The pellets bulged slightly after bonding because of the compression used. They had to be etched in acid thereafter in order to get back to the diameter before bonding.

From the foregoing outline of the bonding process, it was evident that:

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- (a) the compression and the resultant bulge of the previously uniformly-radioactive pellet caused subsequent inhomogeneity in the radioactivity profile of the sample. The rearrangement of zinc-65 around the line of bonding caused more of the activity to 'escape' to the outer radii. Thus when melting occurred, there would be a tendency for the radioactivity to be initially high near the outer radii close to the walls at the immediate vicinity of the radioactive pellet than at the center core. Such a situation could lead to the sharp profiles found near the surface of the pellet in the vicinity of the bonding (Fig. 20 to 22).
- (b) The effect of work hardening of the interface because of bonding could also result in melting and solidifying anomalies which could affect the diffusional process at least initially.

Again recall that the observed radial inhomogeneity occurred close to the bonded zinc pellet and tended to disappear with distance away from the bond line. This was observed in all three samples irrespective of their position from the hot or the cold end of the furnace. These observations tend to confirm the suspicion that the bonding process is to blame.

An obvious check for this theory is to fabricate new zinc samples and test them with a careful monitoring of the zinc-65 distribution immediately after bonding by sectioning it and counting its activity before subjecting it to the melting process. Unfortunately, no funds were provided in this contract for zinc-65 irradiation and fabrication. Experimentation was not provided for.

4. Statistical Test to Check Effect of Scatter of Data on Observed Radial Concentrations

The short computer program, which had been used to evaluate the one-dimensional unsteady state Fick's law of diffusion in the theoretical analysis of the M558 data, was rerun and checked for error. No errors were found and the same results reported in Reference 1 and shown in Fig. 16 to Fig. 18 were obtained again. The experimental data in the axial direction were well modelled by the Fick's law solution.

Next, statistical tests of significance using student t- distribution were made to check whether the calculated data and the experimental data in the axial direction were indeed statistically the same. The result of the statistical test confirmed that the two results were indeed the same. Pairs of data at 25 locations were tested and had been so selected as to cover the entire observed region of the data.

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Similar tests were made in the radial direction to test whether the radioactivity concentration at a given location for the central core (1) was indeed the same as the concentration for the outer section (0). Only six or seven pairs of data points were used in these cases since only six or seven radial data had been obtained for each sample during the sectioning process. At a given percent level of significance, the following equations were used to calculate t for comparison with t in statistical tables:

$$s^2 = \frac{n \sum (d^2) - (\sum d)^2}{n(n-1)} \quad (1)$$

$$t = \frac{O_{avg} - I_{avg}}{s / \sqrt{n}} \quad (2)$$

where

s = standard deviation

d = difference = $O - I$

n = number of pairs of data points

Table 2 illustrates such a calculation made on sample A7 radial data.

TABLE 2

A7 Radial Concentration Data Comparison ($\mu\text{Ci/gm}$)

Inside (1)	Outside (0)	Difference ($d = O - I$)
0.001	0.001	0.000
0.002	0.006	0.004
0.015	0.012	-0.003
0.206	0.181	-0.025
0.796	0.777	-0.019
1.841	1.660	-0.181

$n = 6$, $s = 0.071287$, $I_{avg} = 0.476833$, $O_{avg} = 0.439500$

t (calculated) = - 1.283

Degrees of freedom = $n - 1 = 5$

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At 90% significance level, t (table) = ± 2.015

At 80% significance level, t (table) = ± 1.476

Hence even at 80% significance level, there is no significant difference between inner core concentrations and outer core concentrations.

Table 3 summarizes the results of the statistical tests of significance for each of the three Skylab samples A6, A7 and B5 as tested between inner core concentrations and outer core concentrations.

TABLE 3

SAMPLE	t(calculated)	Degrees of freedom (n-1)	t (table)		Conclusion Drawn
			at 90%	at 80%	
A 6	+ 0.777	6	± 1.943	± 1.440	no difference
A 7	- 1.283	5	± 2.015	± 1.476	no difference
B 5	+ 1.870	5	± 2.015	± 1.476	no difference at 90%

No significant difference could be determined between the concentrations of the inner core and that of the outer core even at 80% level of significance. The only exception is for sample B5 which shows no significant difference at 90% level of significance but at 80% level of significance shows significant difference. This may be the effect of the double bonding process on specimen B. The results of the statistical tests indicated therefore that the observed radial distribution was probably due to data scatter.

CONCLUSIONS

As a result of the present study and review, the following conclusions have been drawn.

1. Statistical tests alone would indicate that no statistically significant radial-concentration inhomogeneity was observed. The observed inhomogeneity could be error due to data scatter.
2. A review of the ORNL data acquisition method showed that it was of high precision and careful accuracy within the equipment constraints at ORNL.
3. The bonding procedure which necessitated the compression of the radioactive pellet and subsequent etching to maintain original diameter could have introduced initial zinc - 65 inhomogeneity by pushing more of it towards the outer walls. Work-hardening of the pellet during the bonding procedure could have also added to the cause of the inhomogeneity. The observed fact that most of the anomalies were observed in the vicinity of the bonded pellets tends to support the foregoing argument.
4. It was possible that surface tension, capillary action near the walls of the porous graphite sleeve and temperature inhomogeneity could have introduced surface perturbations that would result in the radial concentration profiles observed.
5. Detailed experimental and computer model studies could be initiated to evaluate the precise parts played, if any, by the bonding procedure, surface tension and capillary action on the anomalies observed on the M558 radial concentration distribution.

RECOMMENDATIONS

The following action steps are recommended:

1. Until further experimental data are obtained, the observed radial-concentration inhomogeneity of the M558 experiment should be regarded as not of great significance. It could be explained by error due to statistical data scatter.
2. Further ground-based experiments should be performed to determine how the bonding procedure can affect the initial distribution of zinc-65 before it is subjected to a melting process.
3. If an opportunity to fly another Space experiment of this type comes up perhaps in the 1980's, such an opportunity should be utilized to fly an experiment that would try to determine the effects of geometry and surface and temperature phenomena on the observed radial concentrations. Proper design to isolate these possible causes for study could yield a definitive answer to the puzzle.

ACKNOWLEDGMENTS

The author appreciates and acknowledges the careful and precise work done by Dave Braski and Ed Kobisk and others at Oak Ridge National Laboratory. We also acknowledge the important support that Marshall Space Flight Center has given us through these contract awards. Special thanks go to Mr. Charles Schafer of Marshall Space Flight Center for his keen guidance. Finally, the author expresses appreciation to Howard University for permitting him to perform these studies on their premises and with their support.

LIST OF REFERENCES

1. Ukanwa, A. O., "M558 Radioactive Tracer Diffusion", Proceedings Third Space Processing Symposium, Skylab Results Vol. 1, April 30 - May 1, 1974, Marshall Space Flight Center, Alabama 35812.
2. Braski, D.N., and Kobisk, E.H., "Radioactive Tracer Analysis of Flight Samples From Skylab Experiment M558", Contract No. W-74 - 5 - eng-26, ORNL, Oak Ridge, Tenn.
3. Braski, D.N., O'Donnell, F.R., and Kobisk, E.H., "Progress Report on Specimen Fabrication and Ground Tests for NASA Skylab Experiment M558", ORNL TM-4152, Oak Ridge, Tenn., April 1973
4. Nachtrieb, N.H., "Self-diffusion in Liquid Metals", The Properties of Liquid Metals: International Conference at Brookhaven National Lab, Upton, N.Y. 1966, edited by P.O. Adams, H. A. Davies and S. U. Epstein.
5. Leymonie, C. Radioactive Tracers in Physical Metallurgy, John Wiley & Sons, Inc. N.Y., 1963.
6. Jost, W., Diffusion In Solids, Liquids, Gases, 3rd edition, Academic Press, Inc. N.Y., 1960.
7. Curtiss, Leon F., Measurements of Radioactivity, National Bureau of Standards Circular 476, Washington, D.C., Oct. 15, 1949.

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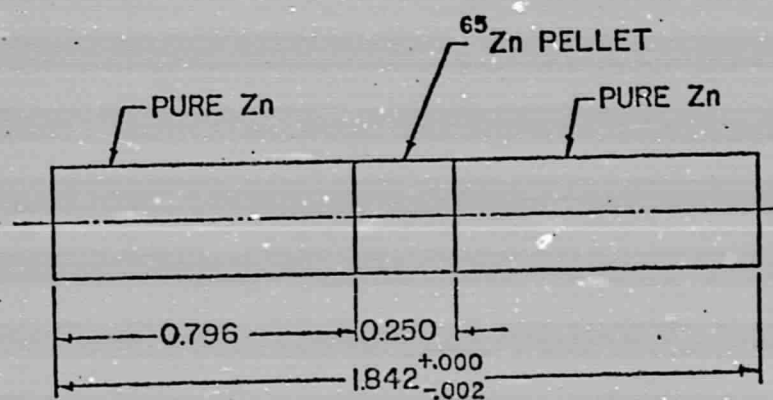
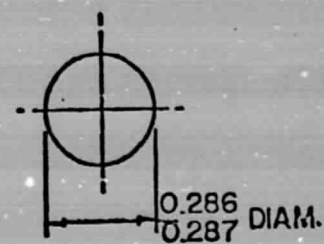
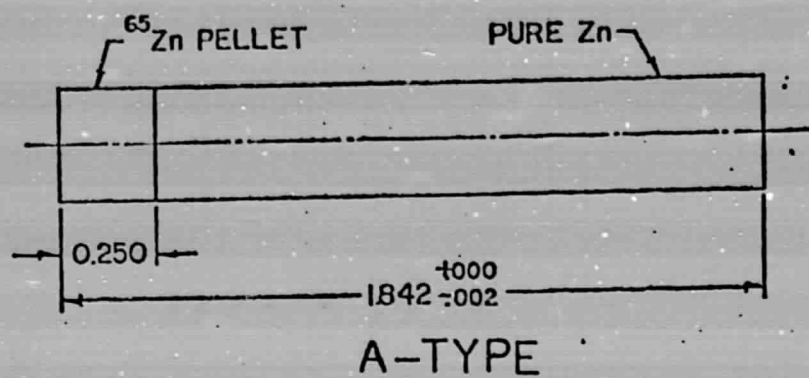
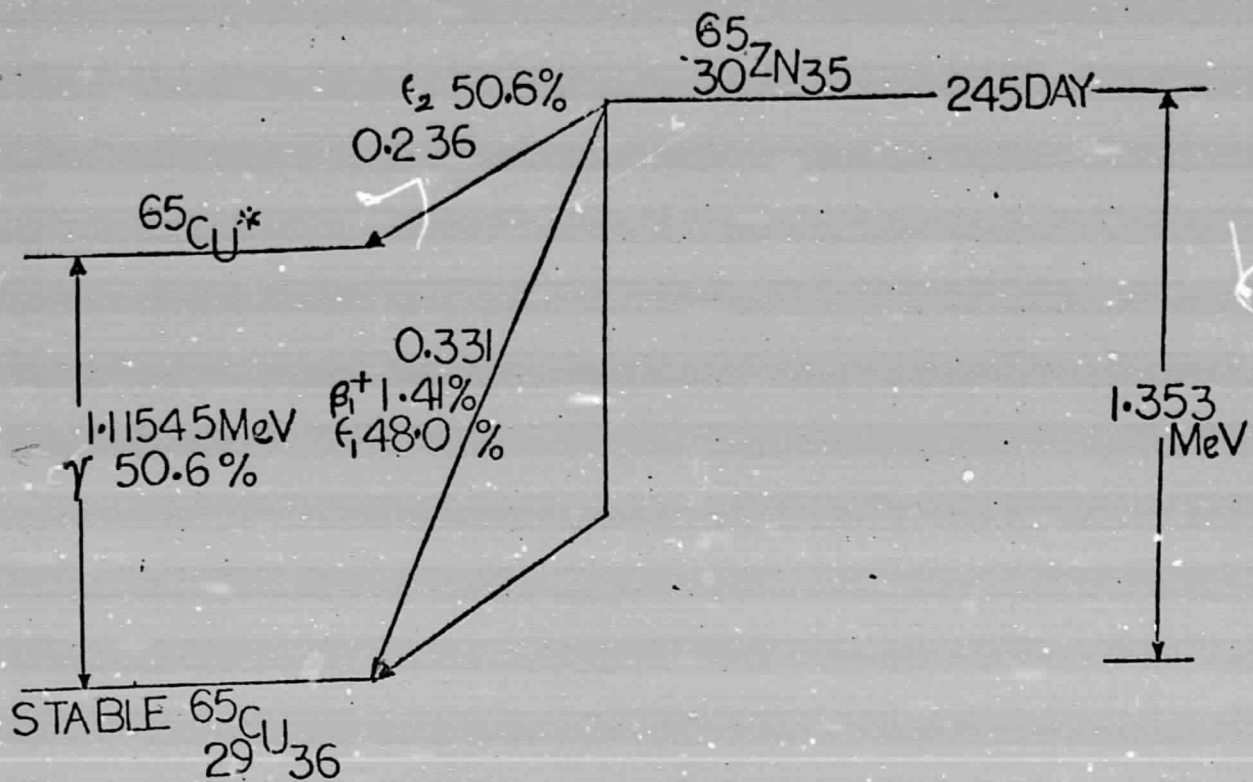


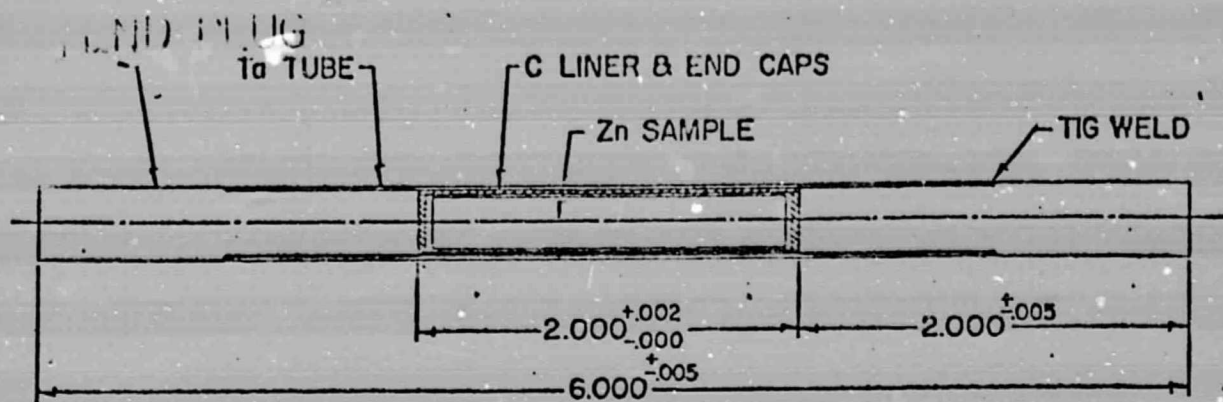
FIG.1 M558 ZINC SAMPLE

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DECAY CONST: 0.0735 DAY^{-1}
 $I(t) = I_0 \cdot \text{EXP}(-0.0735t)$
 DECAY TIME : t DAYS

FIG.2 ^{65}Zn RADIOACTIVE DECAY



DIMENSIONS: INCHES

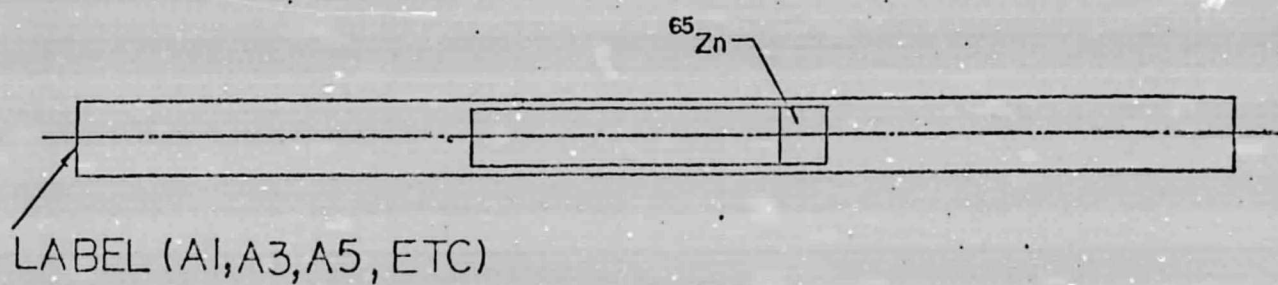
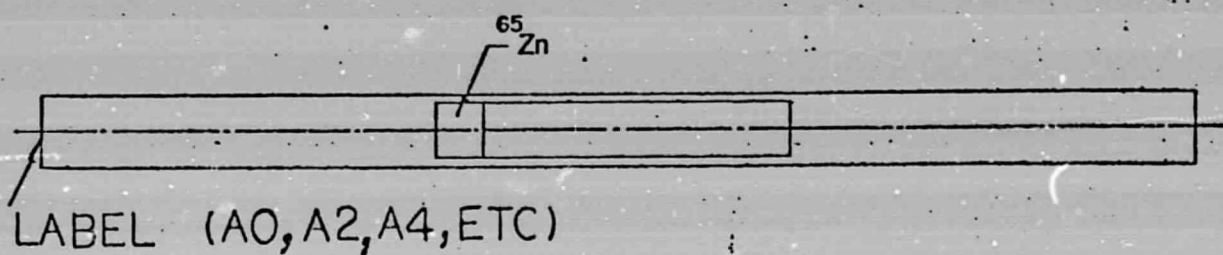
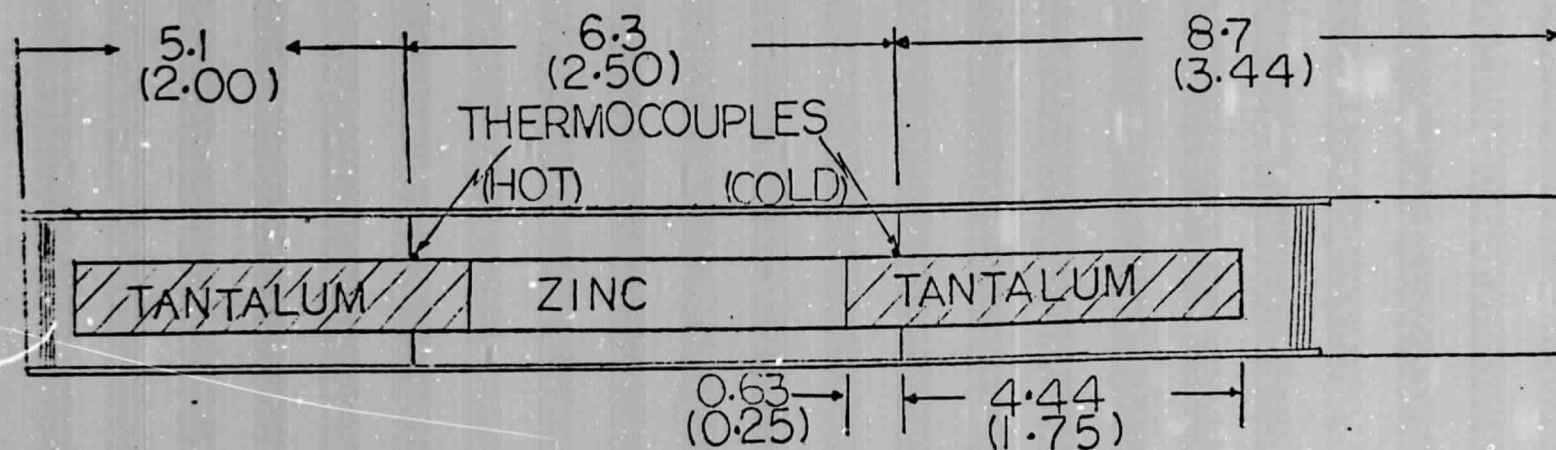


FIG. 3 M558 AMPOULE CONFIGURATION



DIMENSIONS:
CM
(IN.)

M558 PROTOTYPE CARTRIDGE
FIGURE 4

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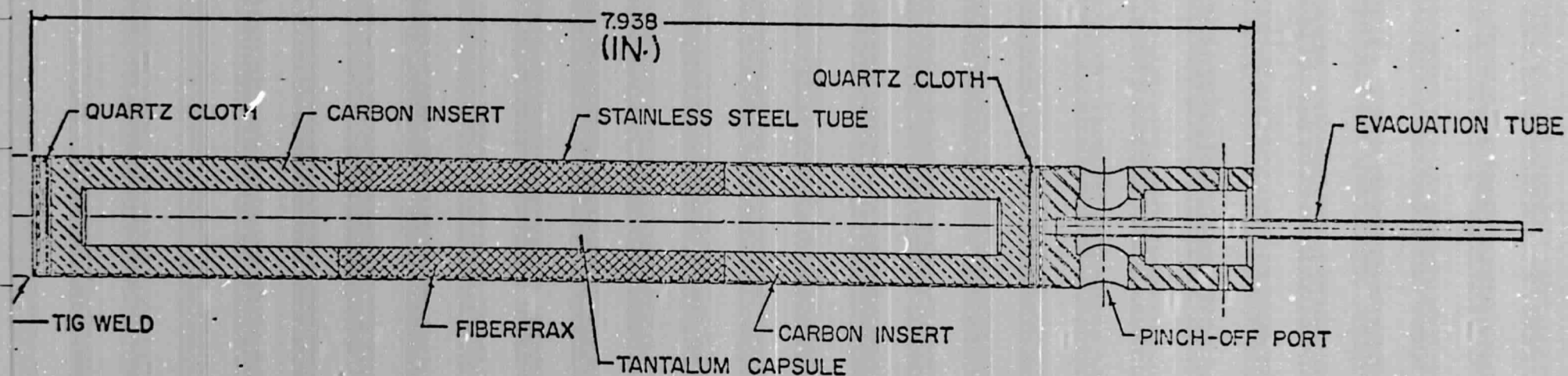
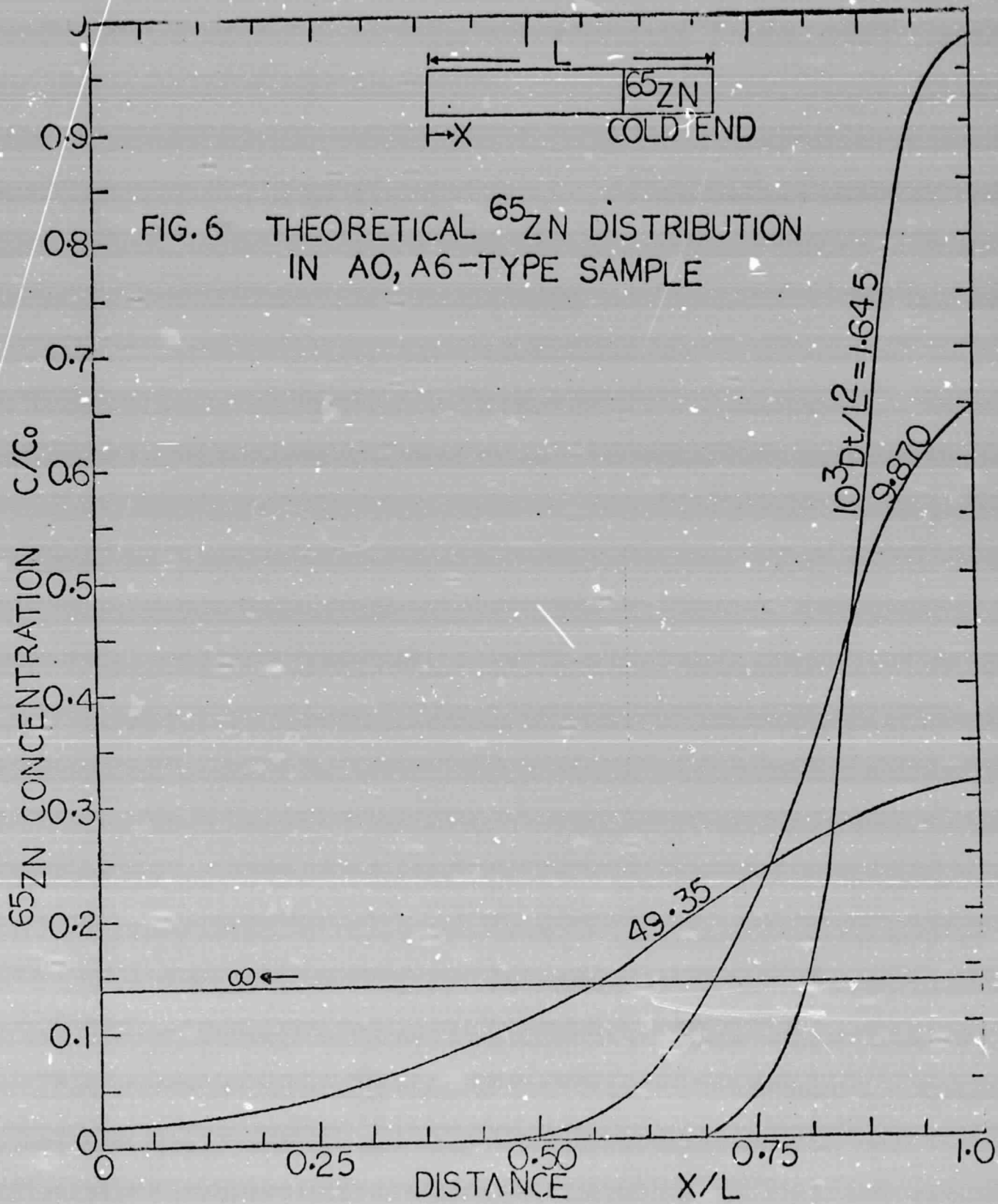
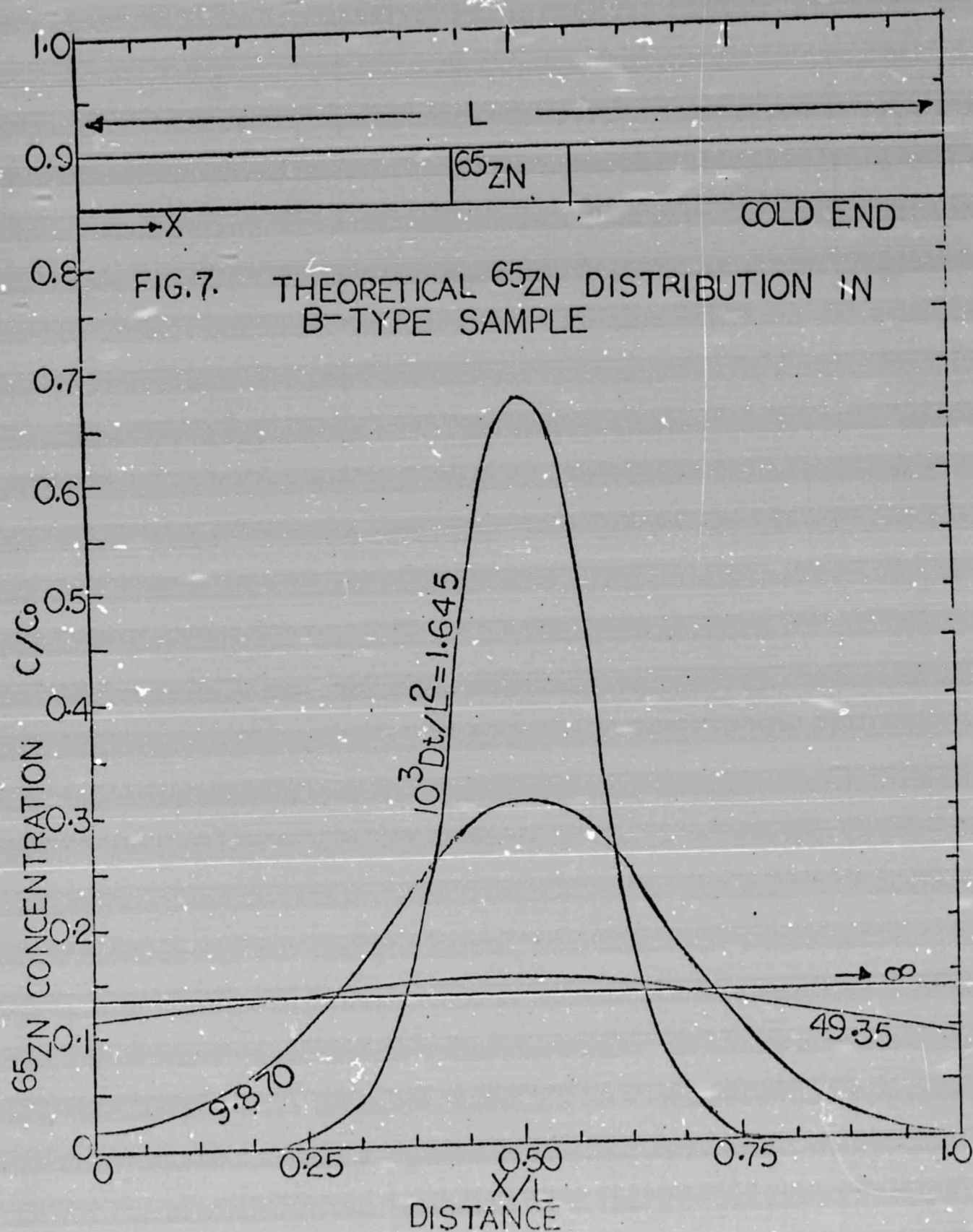


FIG.5

M558 CARTRIDGE

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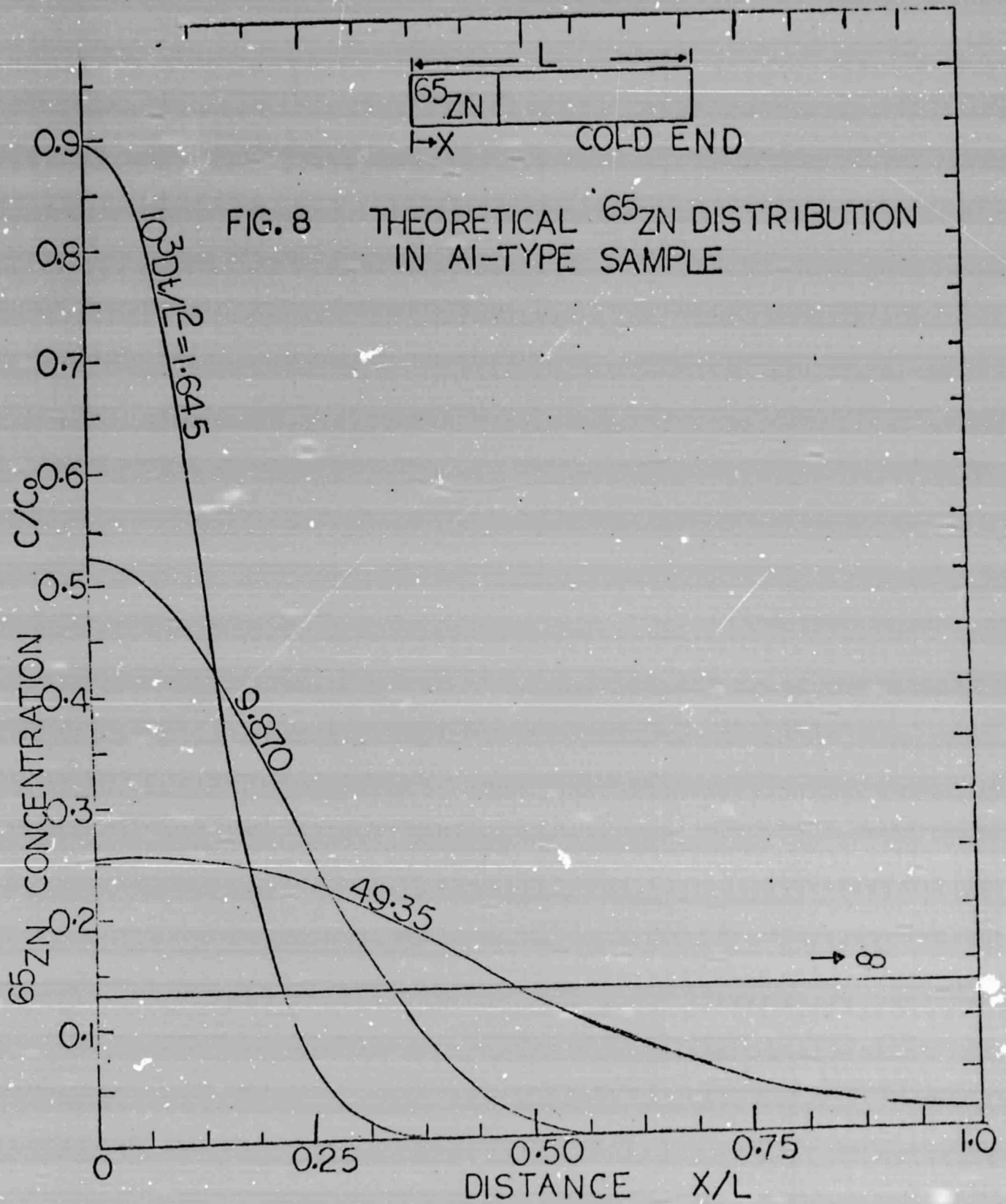


FIG. 9

20

M558 CARTRIDGE STEADY STATE
TEMPERATURE

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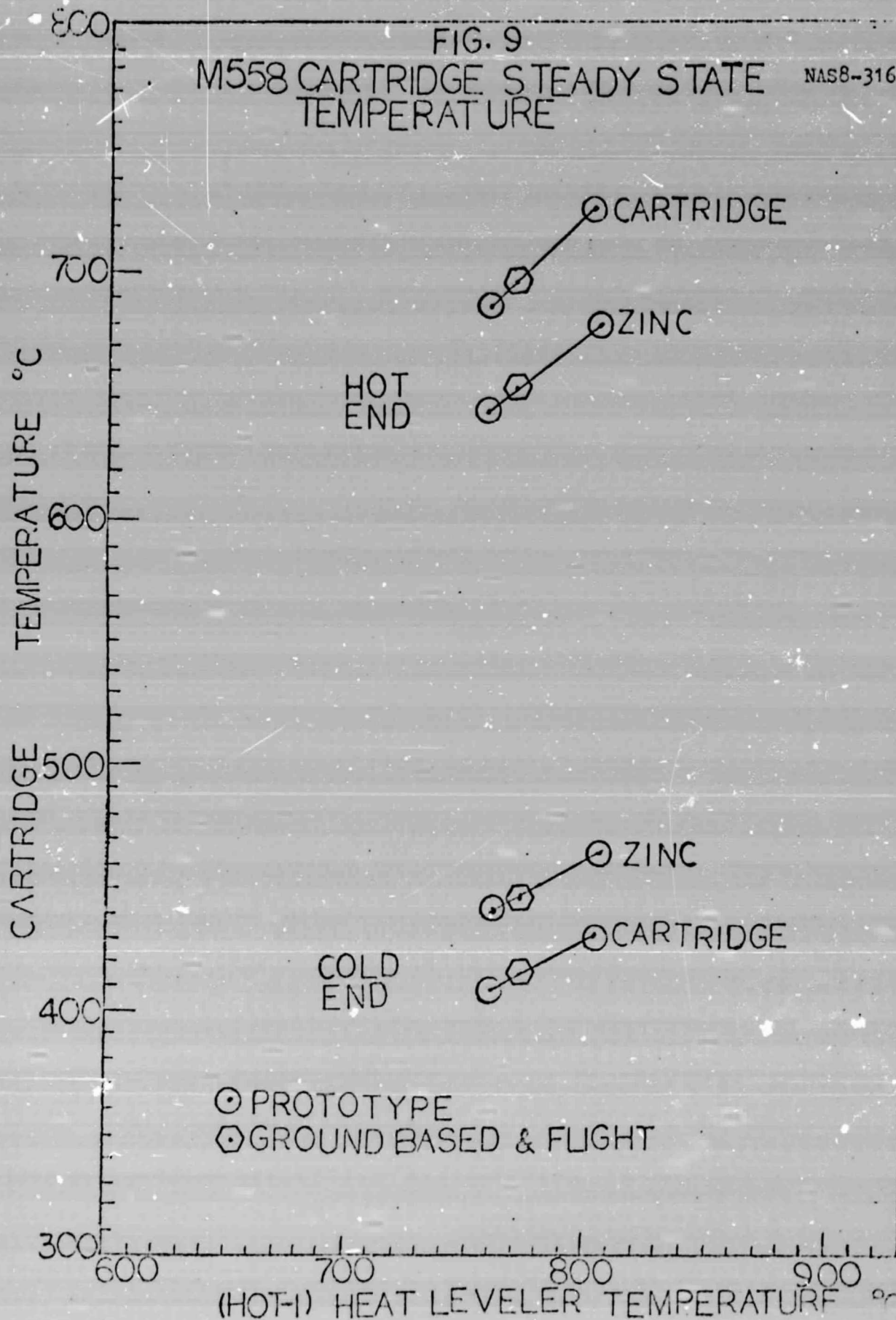
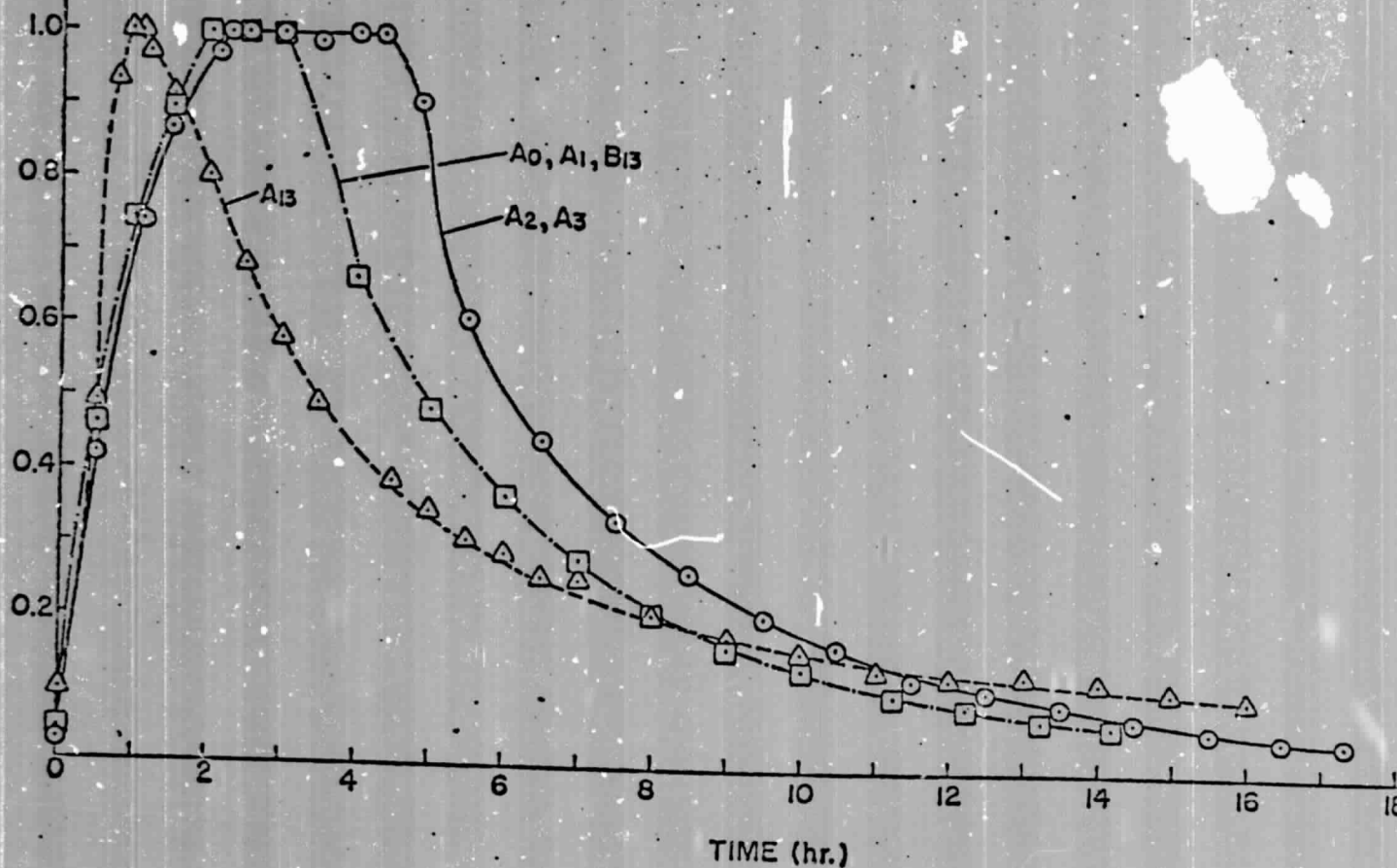
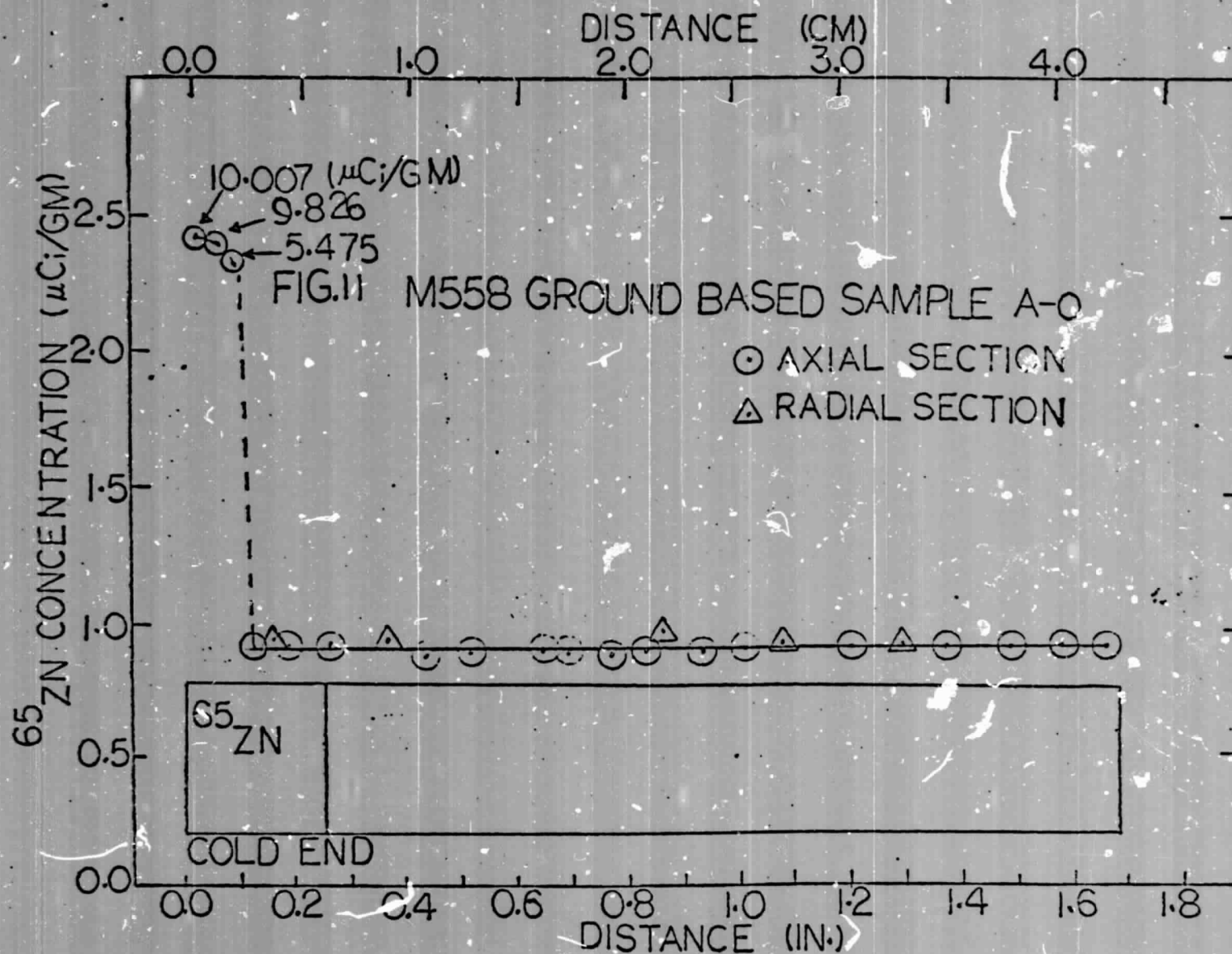
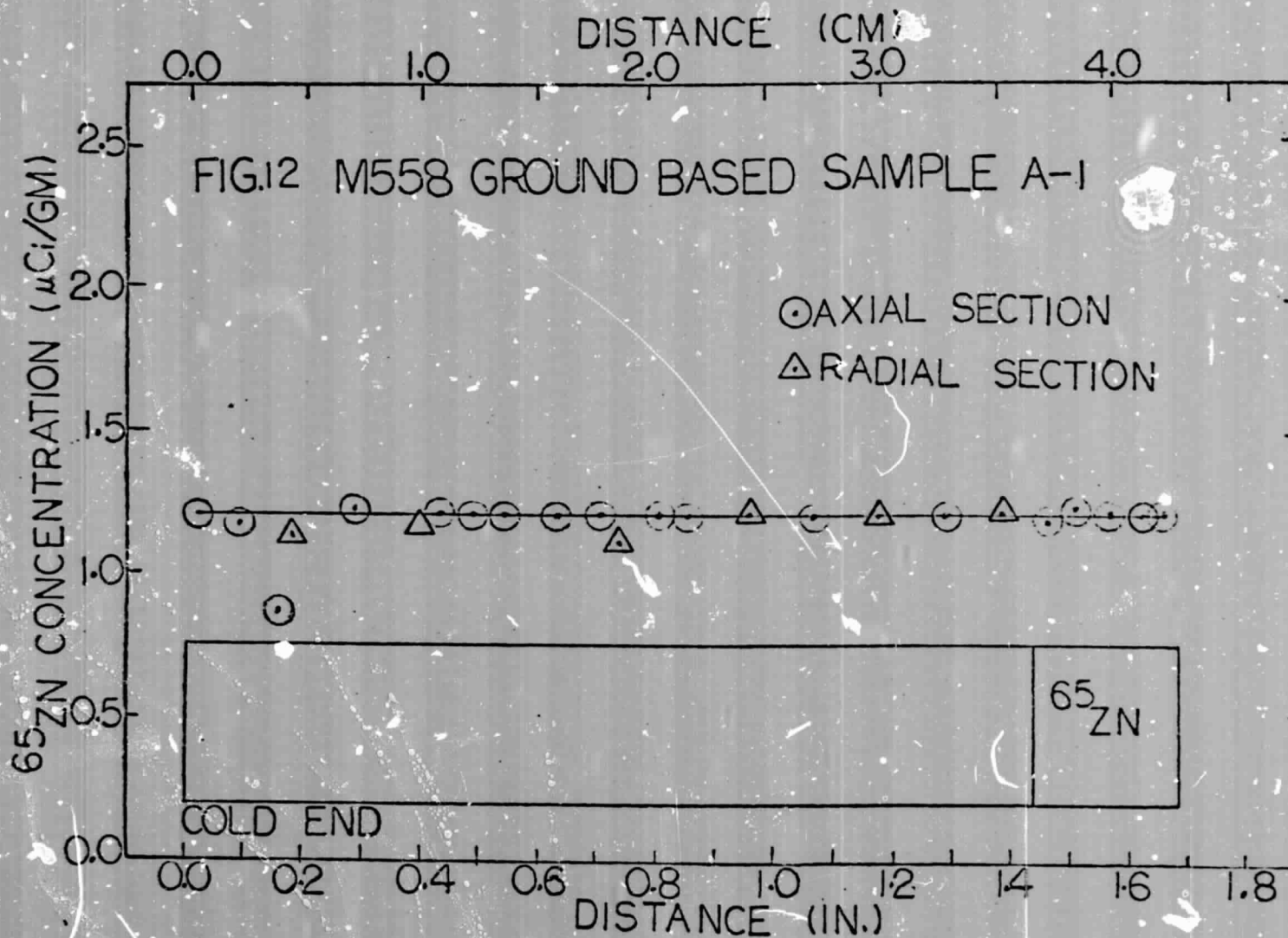


FIG. 10 M558 GROUND-BASED SAMPLE TEMPERATURE

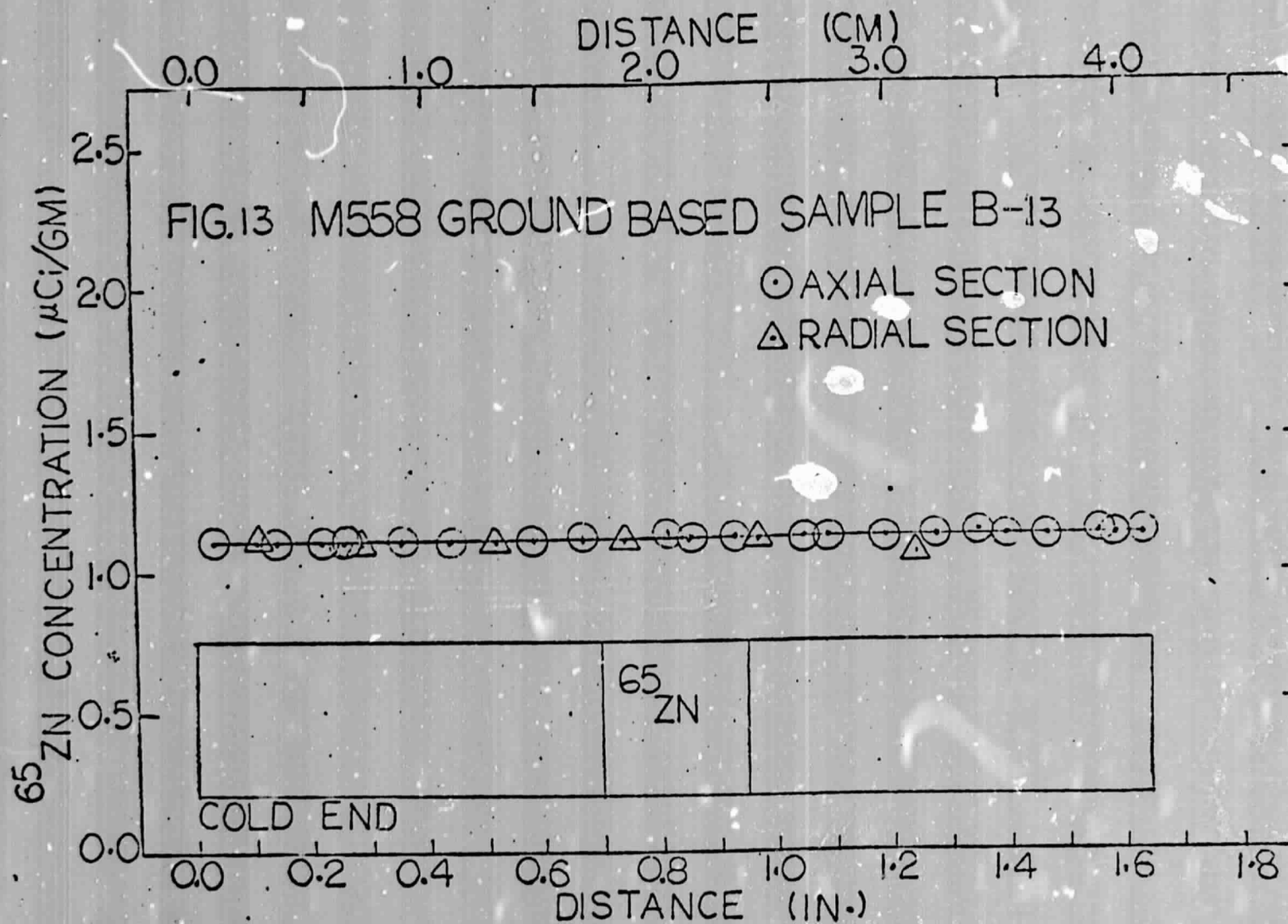
T=CONTROL THERMOCOUPLE TEMPERATURE

 T_{SOAK} = SOAK TEMPERATURE (NASA = 775°C)
(ORNL = 257°C) TO GET TEST GRADIENT





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DISTANCE (CM)

FIG.14 M558 GROUND BASED SAMPLE A-13

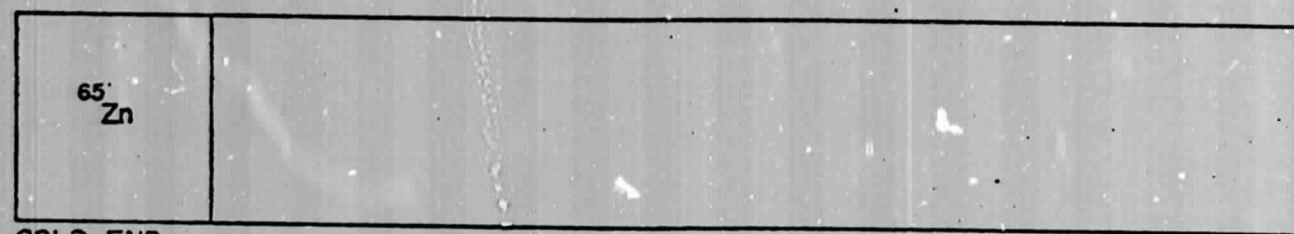
RADIAL SECTION
□ - OUTER
◇ - INNERAXIAL SECTION
○

2.0

1.5

1.0

0.5

 ^{65}Zn CONCENTRATION ($\mu\text{Ci/g}$)COLD END
THERMALLY

DISTANCE (in.)

0

0.2

0.4

0.6

0.8

1.0

1.2

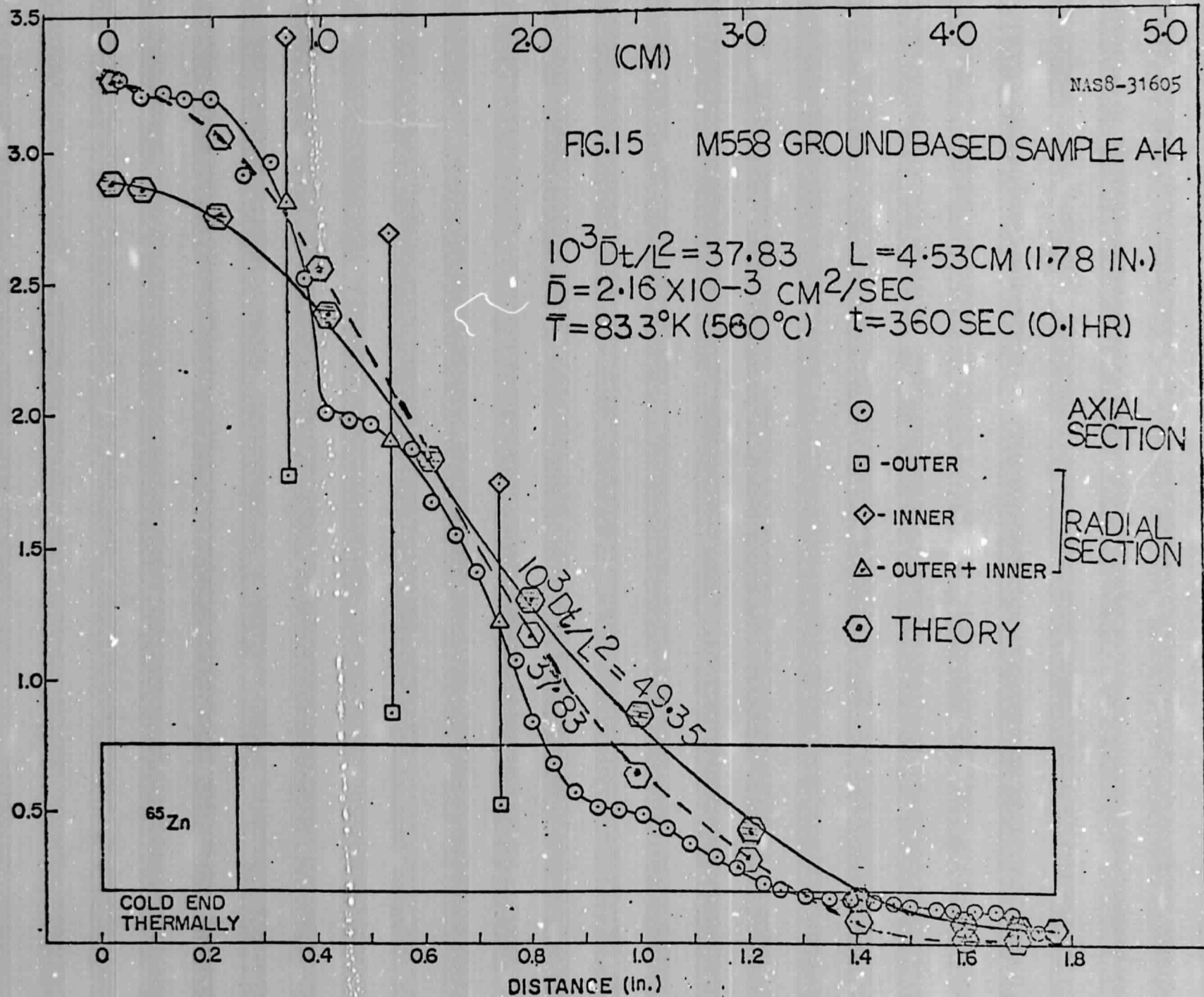
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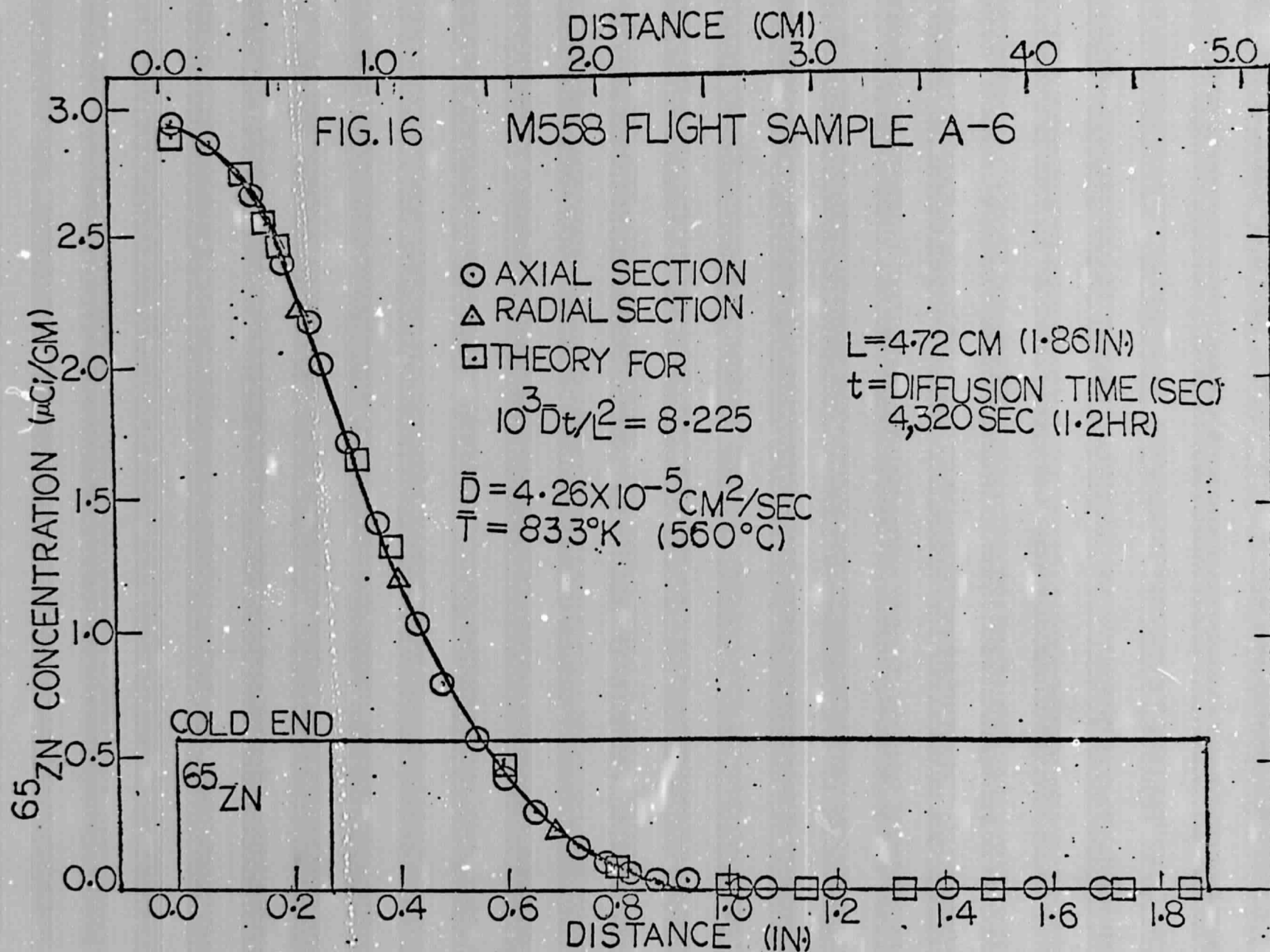
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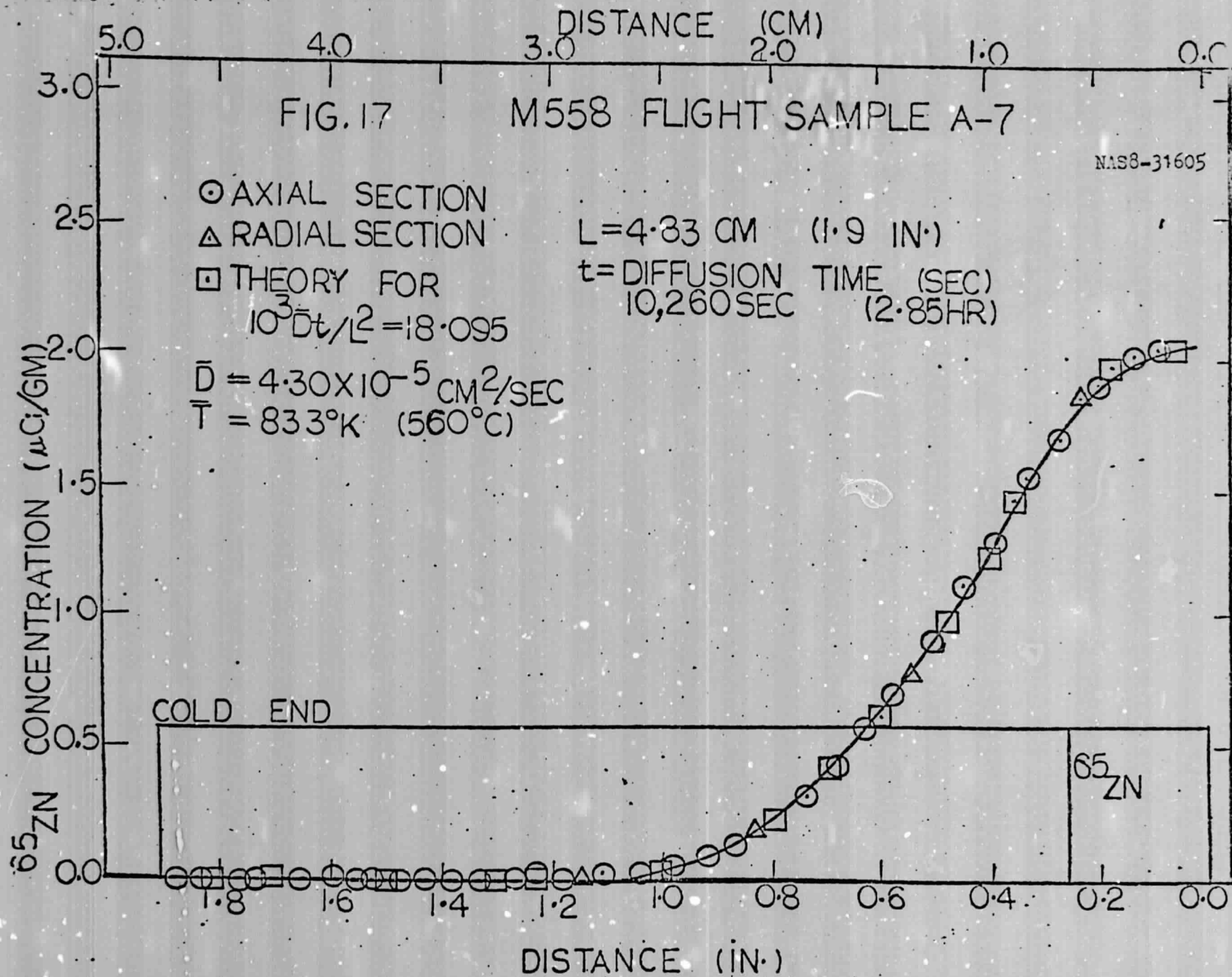
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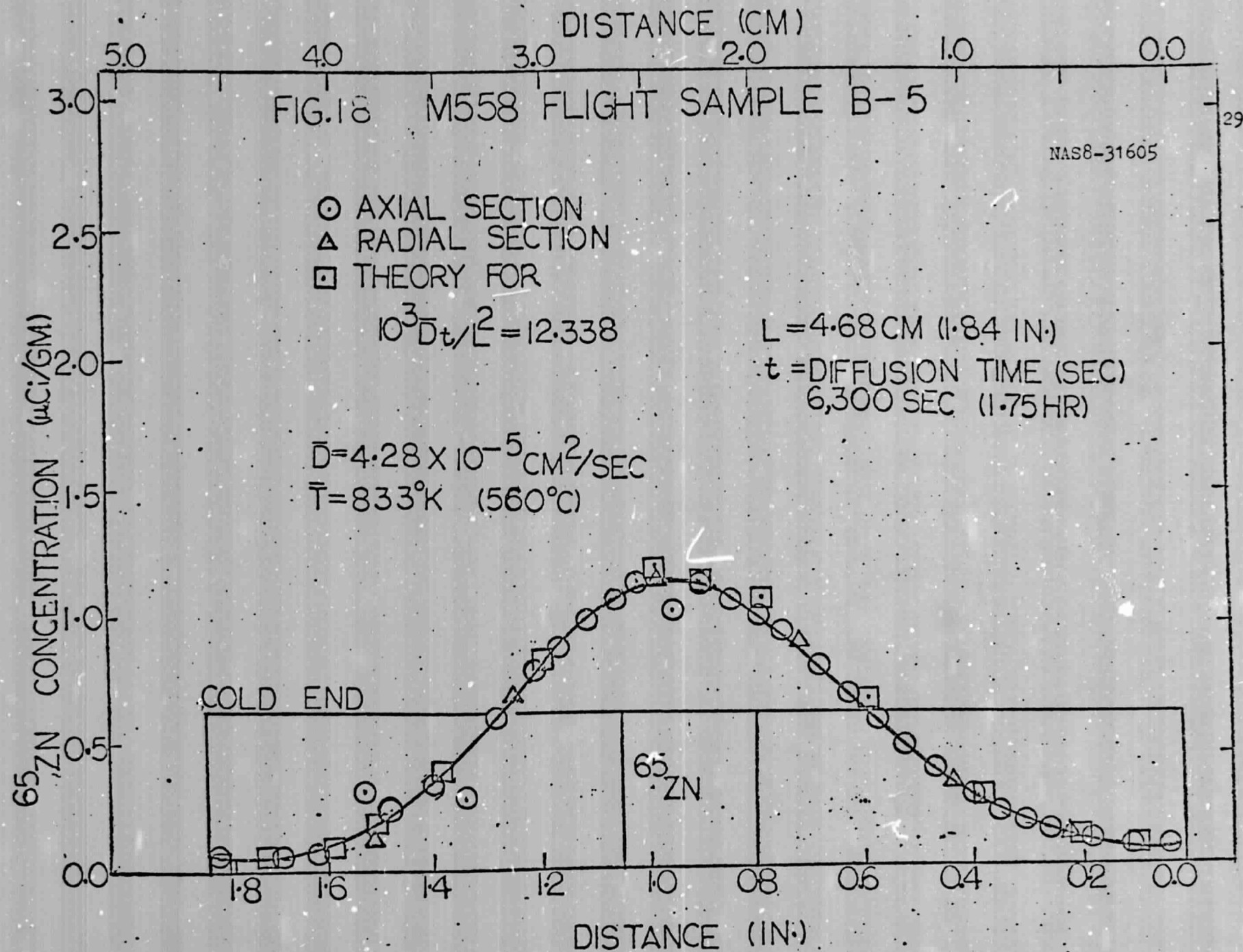
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FIG.15 M558 GROUND BASED SAMPLE A-14

 ^{65}Zn CONCENTRATION ($\mu\text{Ci/g}$)







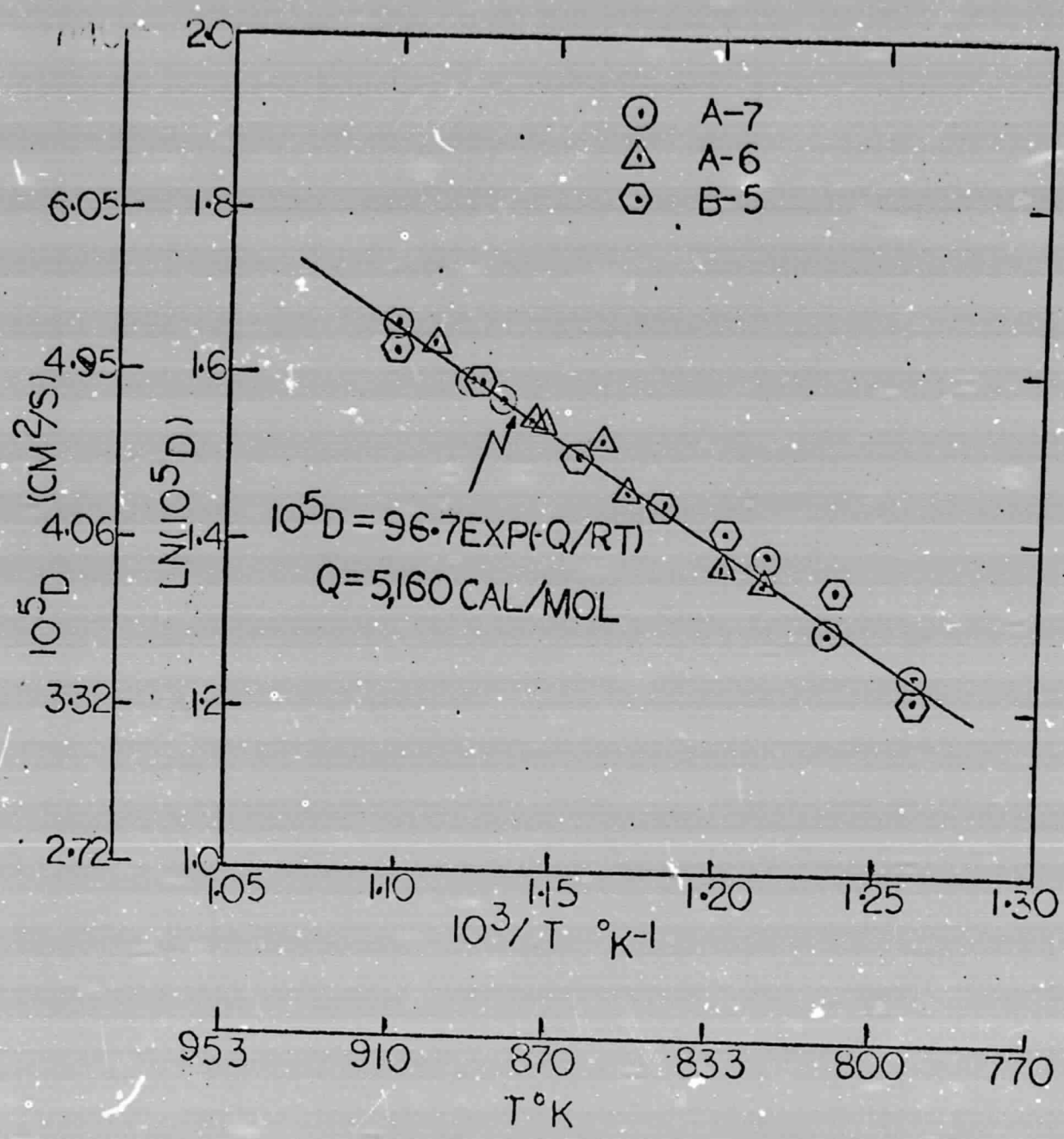


FIG.19 M558 SKYLAB DIFFUSION RESULTS

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FIG.20 M558 FLIGHT SAMPLE A-6
RADIAL ^{65}Zn DISTRIBUTION

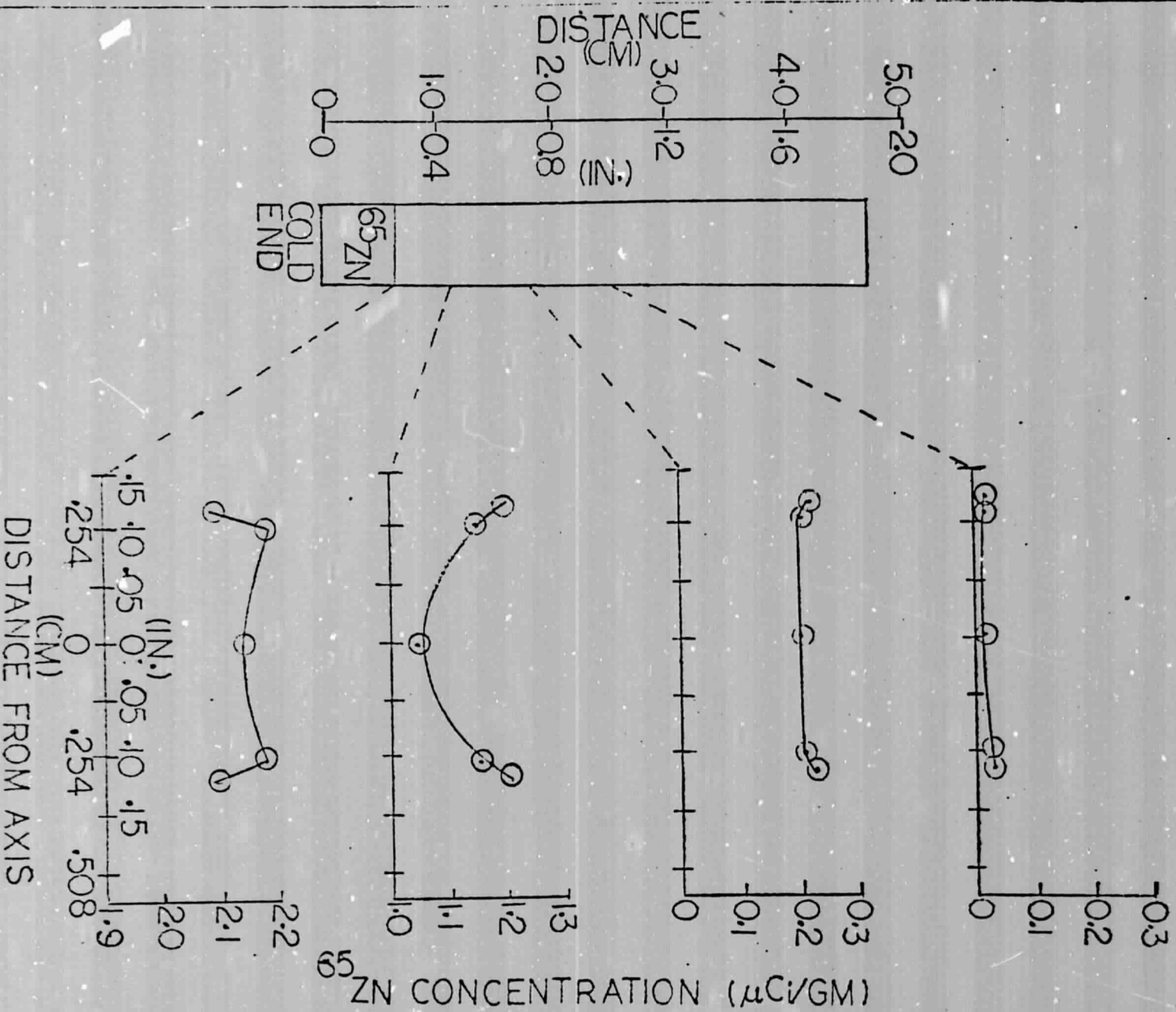


FIG. 21

M558 FLIGHT SAMPLE B-5

RADIAL ^{65}Zn DISTRIBUTION

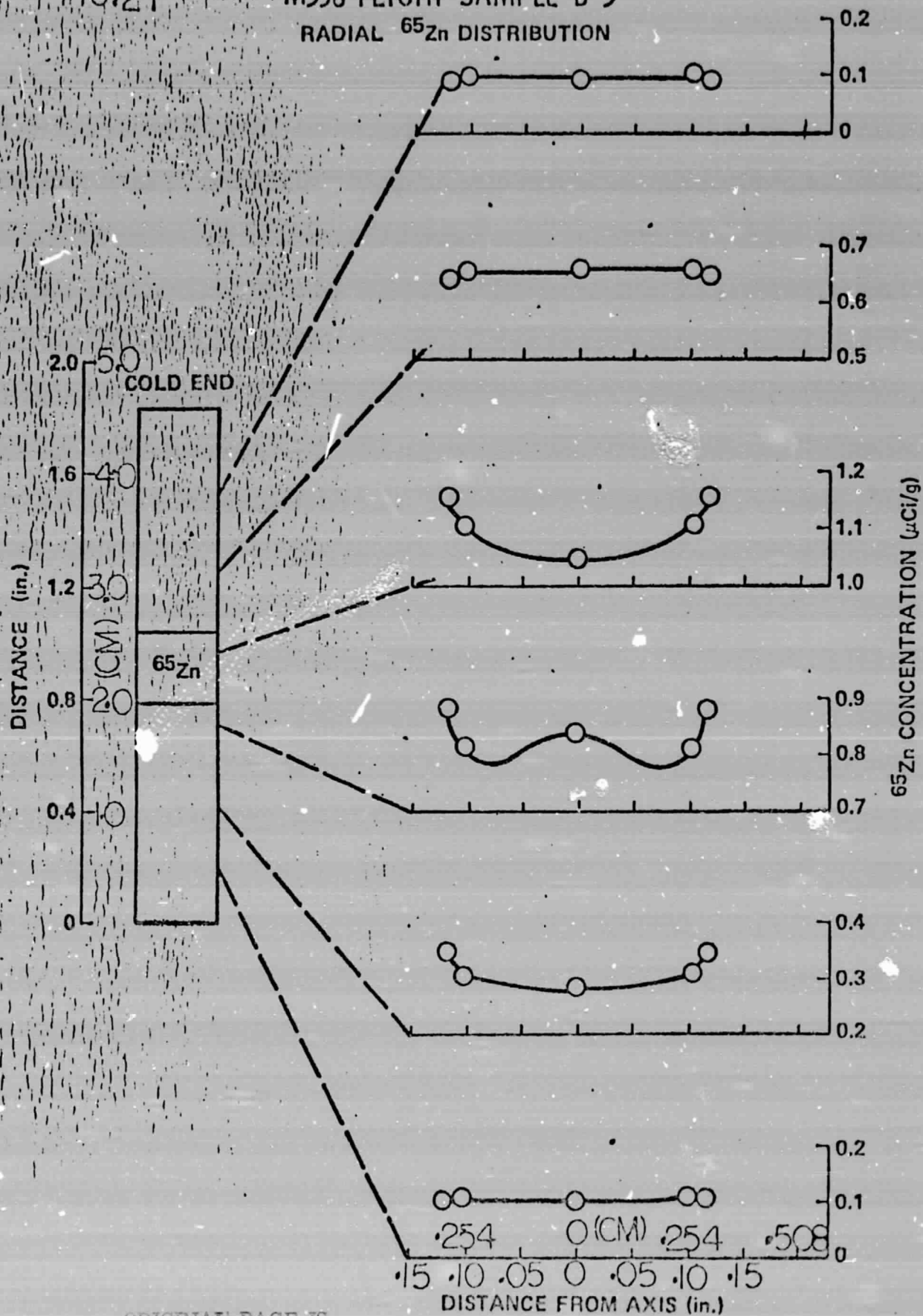
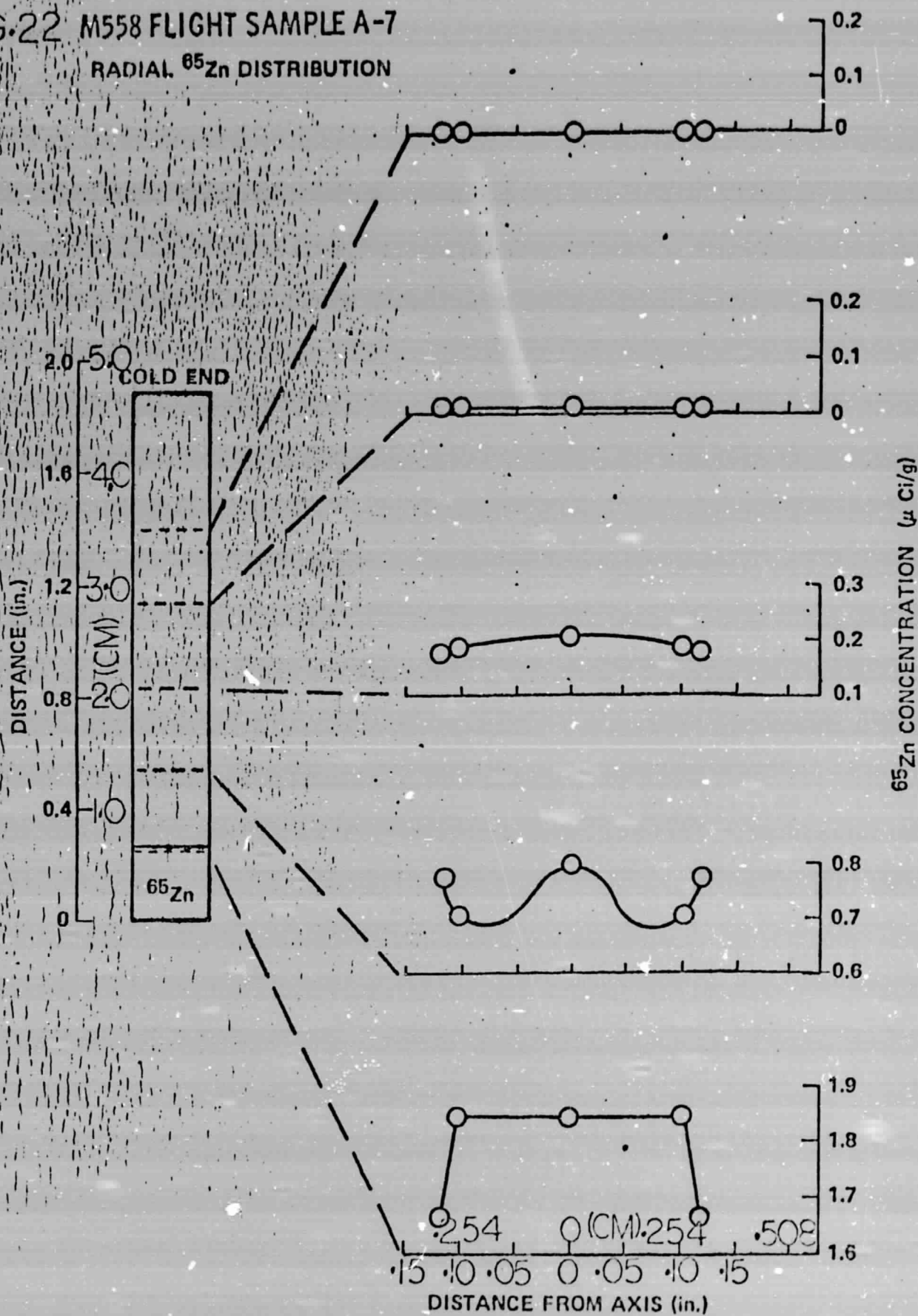


FIG. 22 M558 FLIGHT SAMPLE A-7

RADIAL ^{65}Zn DISTRIBUTION



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